Comprehensive Organic Chemistry Experiments for the Laboratory Classroom

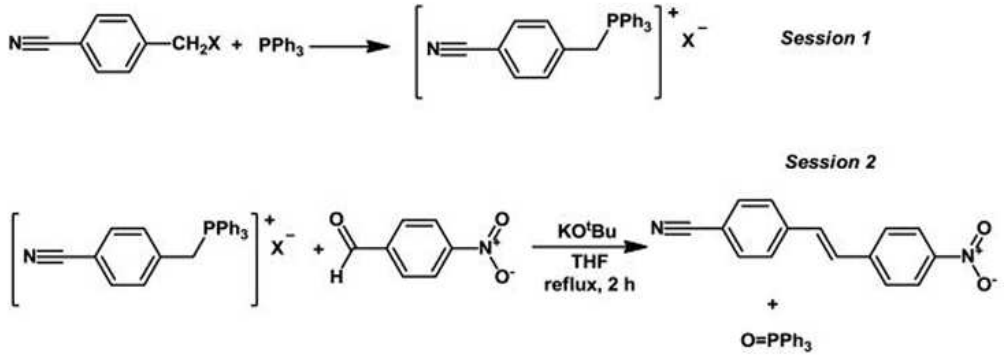
Edited by Carlos A M Afonso, Nuno R Candeias, Dulce Pereira Simão, Alexandre F Trindade, Jaime A S Coelho, Bin Tan and Robert Franzén

ROYAL SOCIETY OF CHEMISTRY

9.2.4. Building an Alkene Spacer by the Wittig Reaction: Synthesis of 4-[2-(4-Nitrophenyl)ethenyl]benzonitrile

António P. S. Teixeira*[a], M. Paula Robalo[b,c], and Paulo J. Mendes[a]

[a]Departamento de Química, Escola de Ciências e Tecnologia, Centro de Química de Évora, Instituto de Investigação e Formação Avançada, Universidade de Évora, Rua Romão Ramalho 59, 7000-671 Évora, Portugal; [b]Área Departamental de Engenharia Química, Instituto Superior de Engenharia de Lisboa, Rua Conselheiro Emídio Navarro, 1, 1959-007 Lisboa, Portugal; [c]Centro de Química Estrutural, Complexo I, Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais, 1049-001 Lisboa, Portugal
*E-mail: apsteix@uevora.pt



Number of sessions (duration of each session)	Hazard level	Difficulty level	Level of study
2 (3 h)	Moderate	Medium	Intermediate

Class names Benzyl halides, phosphorus ylides, aldehydes, alkenes

Concepts involved Wittig reaction, double bond formation, spectroscopic characterization, *E*/*Z* isomers, *E*/*Z* isomeric ratio

Chemicals needed 4-Nitrobenzaldehyde, 4-(bromomethyl)benzonitrile, triphenylphosphine, potassium *tert*-butoxide, toluene, THF, diethyl ether, dichloromethane, methanol, anhydrous magnesium sulfate

Equipment and experimental techniques involved Heating mantle or oil bath with magnetic stirring, Büchner funnel, reflux apparatus, rotary evaporator, structural analysis (NMR, IR, MS)

Keywords Aldehydes, alkenes, phosphorus ylides, precipitation, spectroscopic characterization, Wittig reaction

Background

The Wittig reaction was discovered by George Wittig (Nobel Prize in Chemistry in 1979) in 1954.[1] The Wittig reaction is one of the most common and valuable methods for the formation of alkenes, starting from a carbonyl compound and a primary or secondary alkyl halide. The reaction involves the formation of an alkene from an aldehyde or a ketone using a phosphorous ylide derivative, $R_2C^-\text{-}P^+(C_6H_5)_3$. This latter species is obtained from a nucleophilic substitution reaction of an alkyl halide by triphenylphosphine, followed by deprotonation of the

Comprehensive Organic Chemistry Experiments for the Laboratory Classroom
Edited by Carlos A. M. Afonso, Nuno R. Candeias, Dulce Pereira Simão, Alexandre F. Trindade, Jaime A. S. Coelho, Bin Tan, and Robert Franzén.

Published by the Royal Society of Chemistry, www.rsc.org